भारतीय मानक

Indian Standard

IS 326 (Part 2) : 2023

प्राकृतिक और कृत्रिम सुगंध सामग्री — नमूनाकरण और परीक्षण के तरीके

भाग 2 इत्र सामग्री और नमूनों की प्रारंभिक परीक्षा

(तीसरा पुनरीक्षण)

Natural and Synthetic Perfumery Materials — Methods of Sampling and Test

Part 2 Preliminary Examination of Perfumery Materials and Samples

(Third Revision)

ICS 71.100.60

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April 2023 **Price Group 4**

Fragrance and Flavour Sectional Committee, PCD 18

FOREWORD

This Indian Standard (Third Revision) was adopted by the Bureau of Indian Standards after the draft finalized by the Fragrance and Flavour Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This standard was first published in 1952 entitled as 'Methods of test for essential oils' and subsequently revised in 1968 as 'Methods of sampling and test for natural and synthetic perfumery materials'. Taking cognizance of the need to incorporate instrumental methods of analysis in vogue and to facilitate inclusion of additional test methods or changes in the existing test methods and also to align these test methods with the corresponding ISO Standards, the standard was subsequently revised in 1980 and the committee decided to split the standard and publish individual test methods as separate parts of the original standard.

In this revision, a new method, namely Karl Fischer Method has been introduced for the determination of water content. Also, Colour and Clarity have been removed from Preliminary Examination, since both of them can be detected visually.

In the preparation of this standard, considerable assistance has been derived from BS 2073 : 1976 Methods of test for essential oils. British Standards Institution.

The composition of the committee, responsible for the formulation of this standard is listed in Annex A.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

NATURAL AND SYNTHETIC PERFUMERY MATERIALS — METHODS OF SAMPLING AND TEST

PART 2 PRELIMINARY EXAMINATION OF PERFUMERY MATERIALS AND SAMPLES

(Third Revision)

1 SCOPE

This standard (Part 2) prescribes the special tests for the detection of common impurities and adulterants in natural and synthetic perfumery materials.

2 REFERENCES

The standards listed below contain provisions which, through reference in text constitute provisions of this standard. At the time of publication, the editions indicated were valid. All the standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards listed below:

IS No.	*Title*
IS 326 (Part 21) 2001	:Methods of sampling and test for natural and synthetic perfumery materials: Part 21 Determination of water content — Karl fischer method.
IS 2284 : 1988	Method for olfactory assessment of natural and synthetic perfumery materials

3 TERMINOLOGY

For the purpose of this standard, the definitions of terms given in IS 6597 shall apply.

4 PRELIMINARY EXAMINATION

4.1 General

The original containers selected for sampling and the test samples drawn, as the case may be shall be opened and the condition of the contents noted immediately and reported, particular attention being given to the following.

4.2 By-Note

Report if the material passes a by-note.

NOTE — For detailed olfactory assessment of natural and synthetic perfumery materials, refer to IS 2284. This method is based on a comparison of a given material with its corresponding standard sample.

4.3 Determination of Content of Alcohol Insoluble Solid Impurities

4.3.1 *Outline of the Method*

Extraction of the perfumery material is done with ethanol and weighing of any residue after filtration.

4.3.2 *Reagent*

4.3.2.1 *Ethanol* — 95 percent (v/v)

4.3.3 *Apparatus*

4.3.3.1 *Hot-air-oven* — Capable of being maintained at (105 ± 1) °C

4.3.4 *Procedure*

Weigh accurately about 20 g of the perfumery material into a conical flask. Add about 200 ml of ethanol, mix the content of the flask thoroughly and filter through a sintered glass crucible (porosity 4 is suitable) which has previously been dried in the oven at 105 °C and weighed. Wash any insoluble matter remaining in the flask into the crucible, suck dry and then dry the crucible and its contents in the oven at 105 °C until the loss of mass on drying in the oven for an additional 30 min does not exceed 1 mg.

4.3.5 *Calculation*

Content of alcohol-insoluble solid impurities,

$$\text{Percent by mass} = \frac{(m_3 - m_2)}{m_1} \times 100$$

where

m_1 = mass, in g, of the sample of perfumery material taken;

m_2 = mass, in g, of the empty sintered glass crucible; and

m_3 = mass, in g, of the sintered glass crucible and residue.

4.4 Determination of Water Content

4.4.1 *General*

Two methods are available for determination of the Water Content, namely Oven Drying Method and Karl Fischer Method. In the event of any dispute, Karl Fischer Method may be treated as the Referee Method.

4.4.2 *Oven Drying Method*

4.4.2.1 *Outline of the method*

Distillation of the perfumery materials is done with a water-immiscible solvent.

4.4.2.2 *Reagent*

Heptane — Clear and free from visible water, or toluene.

4.4.2.3 *Apparatus*

4.4.2.1 *Dean and stark apparatus* — With a distillation vessel of 500 ml capacity and a receiver of either 2 ml or 7.5 ml capacity. Clean the apparatus thoroughly with a mixture of potassium dichromate and concentrated sulphuric acid, rinse with clean water and dry before use.

4.4.2.2 *Electric heating mantle or oil-bath*

4.4.3 *Procedure*

Weigh 20 g ± 0.5 g of the perfumery material into the distillation vessel. Half fill the vessel with solvent and add a few dry porcelain chips to regulate boiling. Assemble the apparatus. Maintain the contents of the flask at the boil using the heating mantle or oil-bath until the level of the water layer in the receiver remains constant for 30 min. Cool the apparatus to room temperature and detach any globules of water from the condenser wall by means of the spray tube.

NOTE — Read the volume of water in the receiver.

4.4.4 *Calculation*

Content of water, percent by mass = $\frac{V \times 100\, d}{m}$

where

V = volume, in ml, of water collected in the receiver;

m = mass, in g, of perfumery material taken; and

d = density of water at the air temperature (0.997 1 g/ml at 27 °C).

4.5 Karl-Fischer Method

For the determination of water content by Karl-Fischer Method, refer IS 326 (Part 21) : 2001/ISO 11021 : 1999 ‘Methods of sampling and test for natural and synthetic perfumery materials: Part 21 Determination of water content — Karl Fischer method’.

4.6 Preparation of the Material for Physico-Chemical Analysis

4.6.1 Remove as far as possible any visible water present in the sample by decanting, then add about 10 percent by mass of neutral, freshly ignited and powdered magnesium sulphate and shake the mixture vigorously from time to time during a period of 2 hours. Filter through paper and store if necessary in clean, dry, airtight, opaque, non-absorbent containers, preferably of glass or of metal on which the sample has no action. Fill the sample containers leaving a little air space to allow for expansion and immediately seal after filling. Use only new good quality corks or glass stoppers or screw-lids in the case of glass containers and new good quality corks or sore stoppers in the case of metal containers. To prevent contact with the sample, aluminium foil or other suitable material may be wrapped round corks. Protect samples from heat.

4.6.2 In the case of perfumery materials which are solid or partly solid at room temperature (oils of rose and guaiac wood) warm the material just sufficiently to liquefy it and proceed as above, maintaining the temperature so that it remains liquid throughout.

4.6.3 The colour of some dark coloured perfumery materials may be lightened by shaking them intermittently for 10 min with 1 percent (*m/m*) of tartaric or citric acid. Filter and proceed as above.

ANNEX A

(*Foreword*)

COMMITTEE COMPOSITION

Fragrance and Flavour Sectional Committee, PCD 18

Organization	*Representative*(*s*)
CSIR — Central Institute of Medical and Aromatic Plants, Lucknow	DR PRABODH K. TRIVEDI **(*Chairperson*)**
All India Agarbathi Manufacturers Association, Bengaluru	SHRI SARATH BABU P. S.
Aroma Sales Corporation, New Delhi	SHRI SUNIL KUMAR JAIN
Central Drugs Standard Control Organization, New Delhi	SHRI ASEEM SAHU
Central Drugs Testing Laboratory, Chennai	SHRIMATI C. VIJAYALAKSHMI
Central Revenue Control Laboratory, New Delhi	SHRI SUNEEL MATHUR SHRI PRAFUL DALAL (*Alternate*)
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CKC Fragrance and Flavours Pvt Ltd, Kolkata	SHRI RISHAB KOTHARI SHRI CHANDRAKANT KOTHARI (*Alternate*)
Consumer Voice, New Delhi	SHRI B. K. MUKHOPADHYAY
CSIR — Central Food Technological Research Institute, Mysore	SHRI GIRIDHAR P. SHRI NAGARAJAN S. (*Alternate*)
CSIR — Indian Institute of Integrative Medicine, Jammu	RAJNEESH ANAND
CSIR — Indian Institute of Toxicology Research, Lucknow	DR ALOK DHAWAN SHRI SOMENDU KUMAR ROY (*Alternate* I) DR SHEELENDRA PRATAP SINGH (*Alternate* II)
CSIR — Institute of Himalayan Bio-Resource Technology, Palampur	DR VIJAI KANT AGNIHOTRI
CSIR — Central Institute of Medical and Aromatic Plants, Lucknow	DR SUDEEP TANDON DR CHANDAN S. CHANOTIYA (*Alternate*)
CSIR — North East Institute of Science and Technology, Jorhat	DR SAMIT CHATTOPADHYAY DR MOHAN LAL (*Alternate*)
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Essential Oil Association of India, Delhi	SHRI AJAY K. JAIN SHRI PRADEEP KUMAR JAIN (*Alternate*)
Fab Flavours and Fragrances Pvt Ltd, Delhi	SHRI GURNISH SINGH

Organization	*Representative*(*s*)
Forest Research Institute (FRI), Dehradun	DR V. K. VARSHNEY
Fragrance and Flavour Development Centre, Kannauj	SHRI S. V. SHUKLA SHRI NADEEM AKBAR (*Alternate*)
Fragrances and Flavours Association of India, Mumbai	SHRI RISHABH C. KOTHARI SHRI JAIDEEP GANDHI (*Alternate*)
Givaudan India Pvt Ltd, Mumbai	SHRI AJIT PAL SHRI ARSHDEEP K JOSHI (*Alternate*)
Indian Beauty and Hygiene Association, Mumbai	MS MALATHI NARAYANAN DR MANAS V. VYAS (*Alternate*)
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Jagat Aroma Oils Distillery, Kannauj	SHRI PRADEEP KAPOOR SHRI VIBHU KAPOOR (*Alternate*)
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Lalji Aromatics Pvt Ltd, Lucknow	SHRI RAHUL MEHROTRA
MSME Testing Center, New Delhi	SHRI MANOJ KUMAR SHRI VIPUL GAIKWAD (*Alternate* I) SHRI L. B. S. YADAV (*Alternate* II)
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Organization	*Representative*(*s*)
Voluntary Organization in Interest of Consumer Education (VOICE), New Delhi	SHRI M. A. U. KHAN
In Personal Capacity (*Flat No. 201, Vishakha CHS Ltd, off, Swami Netyanand Marg, Near Gokhale Bridge, Andheri East — 40006*)	SHRI G. S. RANADE
BIS Director General	SHRIMATI MEENAL PASSI, SCIENTIST 'F'/SENIOR DIRECTOR AND HEAD (PETROLEUM, COAL AND RELATED PRODUCTS) [REPRESENTING DIRECTOR GENERAL (*Ex-officio*)]

Member Secretary
SHRI SOURAV MONDAL
SCIENTIST 'B'/ASSISTANT DIRECTOR
(PETROLEUM, COAL AND RELATED PRODUCTS), BIS

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